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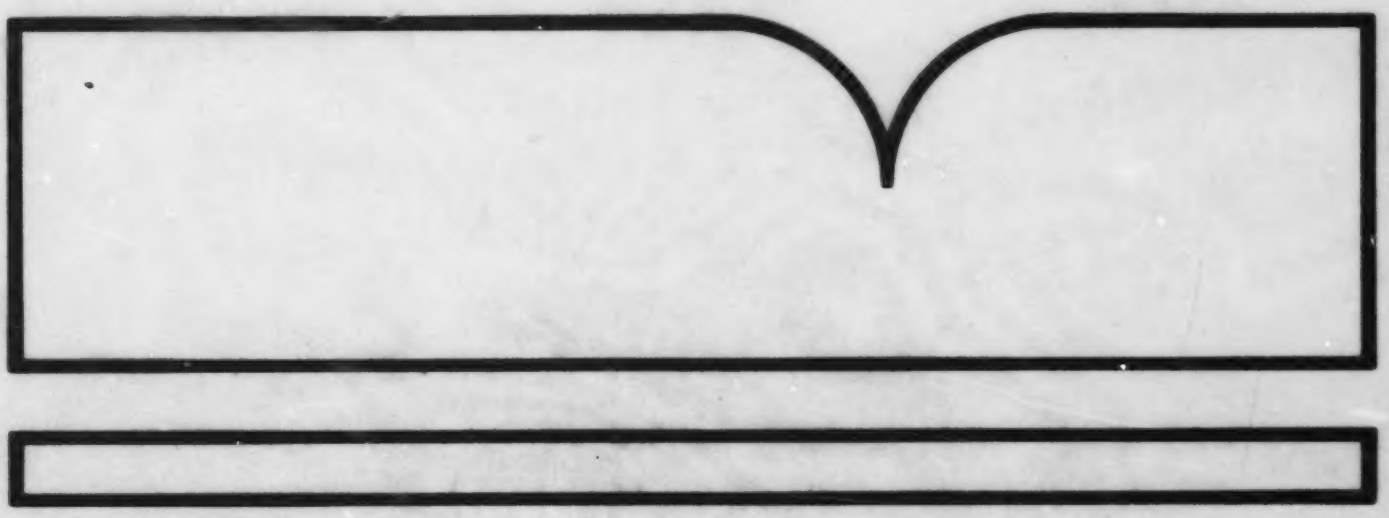
Evaluation of Molecular Sieves for Sampling NOX Emissions at Electric Utility Plants

SouthWest Research Inst.
San Antonio, TX

Prepared for

Environmental Monitoring Systems Lab.
Research Triangle Park, NC

Apr 81



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A field study was conducted to evaluate the use of molecular sieves for collection of NO_x ($NO + NO_2$) emissions from utility boilers. It was found that NO_x could be collected efficiently (99%) at sample flow rates of 1 L/min over 20- to 40-min sampling periods at the NO_x levels encountered in modern utility boilers. The removal of the stack gas moisture by use of an impinger train or condenser ahead of the sample train was shown to be necessary for efficient NO_x collection.

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EVALUATION OF MOLECULAR SIEVES FOR SAMPLING NO_x EMISSIONS AT ELECTRIC UTILITY PLANTS

by

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FOREWORD

Measurement and monitoring research efforts are designed to anticipate potential environmental problems, to support regulatory actions by developing an in-depth understanding of the nature and processes that impact health and the ecology, to provide innovative means of monitoring compliance with regulations, and to evaluate the effectiveness of health and environmental protection efforts through the monitoring of long-term trends. The Environmental Monitoring Systems Laboratory, Research Triangle Park, North Carolina, has responsibility for: assessment of environmental monitoring technology and systems; implementation of agency-wide quality assurance programs for air pollution measurement systems; and supplying technical support to other groups in the agency including the Office of Air, Noise and Radiation, the Office of Toxic Substances, and the Office of Enforcement.

This work was carried out as part of a study to develop new methods for sampling and analysis of NO_x in power plant emissions.

Thomas R. Hauser

Thomas R. Hauser, Ph.D.
Director
Environmental Monitoring Systems Laboratory
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ABSTRACT

A field study was conducted to evaluate the use of molecular sieves for collection of NO_x ($NO + NO_2$) emissions from utility boilers. It was found that NO_x could be collected efficiently (>99%) at sample flow rates of 1 L/min over 20- to 40-min sampling periods at the NO_x levels encountered in modern utility boilers. The removal of the stack gas moisture by use of an impinger train or condenser ahead of the sample train was shown to be necessary for efficient NO_x collection.

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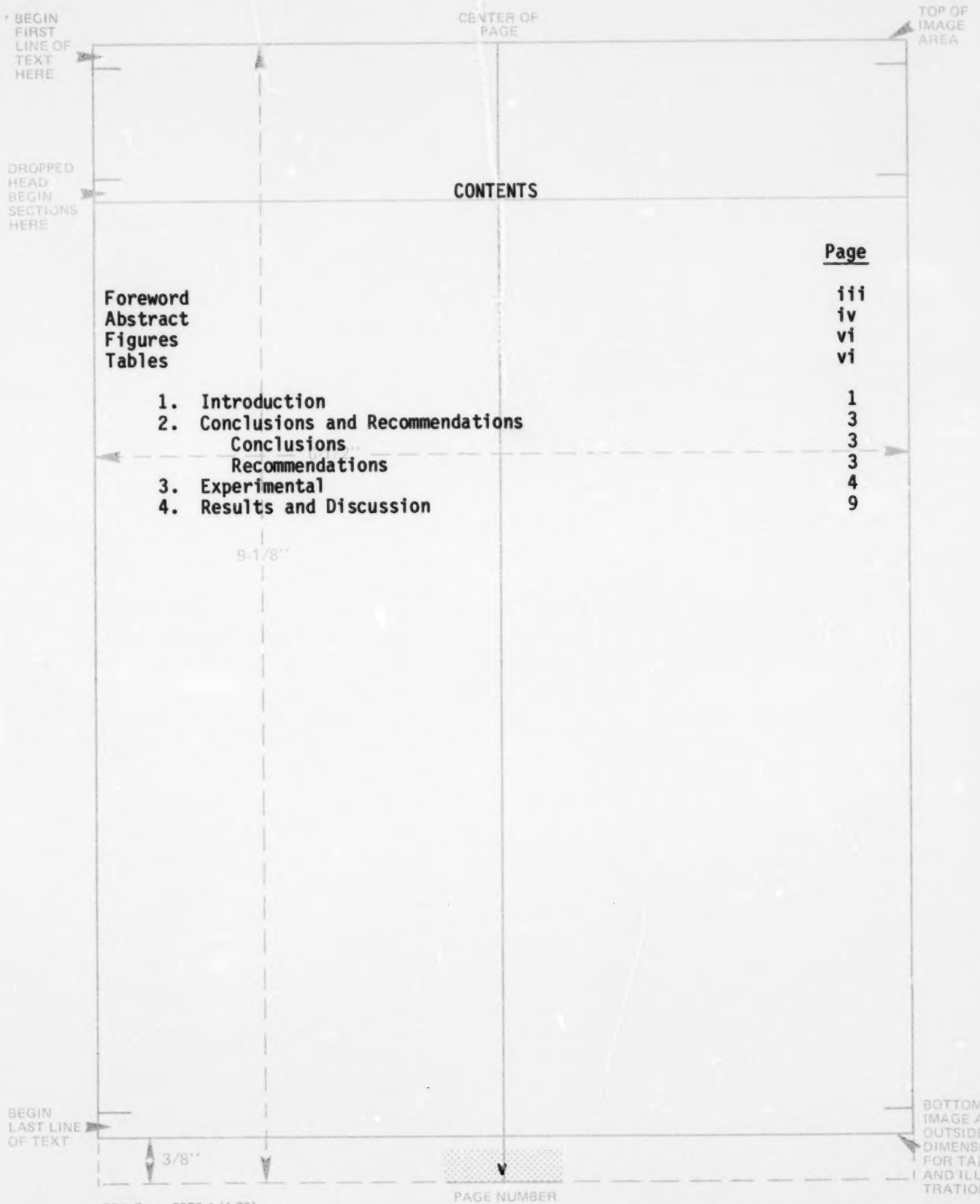

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SECTION 1

INTRODUCTION

The Source Branch (SB) of the Quality Assurance Division (QAD), EMSL/EPA, is involved in developing new methods for the measurement of NO_x emissions from electric utility plants. The main reason for undertaking this work is that the present reference method (Method 7) for measuring these emissions is restricted to a sampling time of only 15 seconds. This then necessitates multiple samplings to obtain a sample representative of the emissions.

The initial phase of this work involved a laboratory study at SB to screen potential NO_x collection systems. (This work will be published at a later date.) During the course of this study, an experiment was conducted in which chromium trioxide supported on molecular sieves was evaluated to determine if NO_x could be quantitatively oxidized to NO_2 and adsorbed on the chromium trioxide. A parallel experiment using only molecular sieves was run as a control. While the desired oxidation did not occur, it was found that the molecular sieves retained the NO_x with good efficiency. Further evaluation in the laboratory was conducted to assess the collection efficiency of various molecular sieves for NO_x collection. A work assignment was then issued to Southwest Research Institute to conduct a field study to assess the feasibility of collecting NO_x emissions from a coal-fired electric utility plant stack gas using molecular sieves.

The field sampling train utilized was similar to an EPA Method 6 sampling train with the following exceptions: the molecular sieve containers (either a 2.0 x 14-cm glass tube, glass midget impingers, or stainless steel midget impingers) were used in place of the standard midget impinger setup of Method 6, and provisions were made to monitor the effluent from the molecular sieve bed with a continuous NO_x monitor to assess NO_x collection efficiency.

During sample collection, all standard sampling practices were employed. The probe was fitted with a glass wool plug to remove particulate matter ahead of the train. Pre- and post-sampling leak checks of the

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sample train were conducted for each run. The continuous NO_x monitor was periodically checked for zero and span calibration. Span calibration was conducted using an NBS traceable gas standard (NO in nitrogen).

The variables studied were: 1) the type and amount of molecular sieve used; 2) the type of molecular sieve container used; 3) the effect of sample flow rate on NO_x absorption efficiency; 4) the effect of uncondensed water vapor in the sample on NO_x collection efficiency; 5) the effect of cooling the molecular sieve bed on NO_x collection efficiency; and 6) effect of the NO_x concentration in the stack gas on NO_x collection efficiency. This latter variable could not be studied systematically. However, due to the fact that the NO_x levels in the stack gas were found to vary as a function of the generating load and the boiler firing conditions, the collection efficiency of the molecular sieves was evaluated over a range of 195 to 350 ppm NO_x.

The NO_x-molecular sieve samples generated in this work will be used by the SB to test and refine analytical recovery procedures developed in the laboratory.

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SECTION 2

CONCLUSIONS AND RECOMMENDATIONS

CONCLUSIONS

The data obtained in this study indicate that molecular sieves provide a sampling medium for high efficiency (>99%) collection of NO_x from fossil fuel-fired utility boilers.

Sample collection efficiency is directly proportional to the amount of molecular sieve used in the sampling train and inversely proportional to the sample flow rate. Sampling can be carried out for 40 minutes at a flow rate of 1.0 L/min with 60 g of molecular sieves with a 99+% collection efficiency at the NO_x levels encountered in this study.

The stack gas moisture content must be removed ahead of the molecular sieves. Failure to remove the moisture rapidly depletes the capacity of the sieves for NO_x due to absorption of the water vapor.

RECOMMENDATIONS

Since the moisture removal system showed some NO absorption, other moisture removal systems, such as cold traps, should be investigated in order to minimize NO_x absorption. Conversely, the present moisture removal system can be used and the contents analyzed for NO_x.

Further testing should be conducted on the molecular sieve collection procedure at different fuel-type boilers and after different type control devices.

Further work is needed to complete development of the cadmium reduction-colorimetric and nitrate-electrode-analytical recovery procedures at SB before a viable sampling and analytical procedure can be specified.

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SECTION 3

EXPERIMENTAL

The field sampling was conducted at the J. T. Deeley Power Plant operated by the City Public Service Board, San Antonio, Texas. A schematic of the sampling train used is shown in Figure 1. In this initial study, three types of molecular sieves were utilized (Linde 13X, Linde AW-500, and Davison Grade 626). The characteristics of the molecular sieves are: Linde 13X, 1/16-inch pellets, 10Å pore size; Linde AW-500, 1/16-inch pellets, 5 Å pore size; Davison Grade 626, 8-12 mesh beads, 5 Å pore size, high crystallinity. The variables studied were geometry of the molecular sieve container (either a vertically mounted 2 x 14-cm glass tube or a glass midget impinger), effect of drying the sample prior to NO_x collection, and the effect of cooling the molecular sieves, in addition to the type of molecular sieve used. The Dynasciences NO_x monitor was calibrated prior to the testing and checked periodically during the testing, using an NBS-traceable gas standard (NO in nitrogen). Stack gas NO_x concentration was determined before and after each run using the monitor; the molecular sieve bed effluent was monitored during the run and the effluent NO_x concentration recorded at two-minute intervals.

The second field sampling study was conducted using the same sampling train. The variables studied included the amount of molecular sieve used (two versus three midget impingers containing 19.6 g of sieve each), the effect of sample flow rate on NO_x collection efficiency, and the effect of cooling the sieve bed on collection efficiency. All runs were made with a water vapor knockout ahead of the molecular sieves. The molecular sieve-containing impingers were cooled in an ice-water bath. The effect of cooling the sieve bed was determined by using either glass or stainless steel midget impingers. An auxiliary experiment was conducted in which samples of stack gas and the calibration standard were taken in Tedlar sample bags at the test site for subsequent analysis in the laboratory using a ThermoElectron Model 10A chemiluminescent

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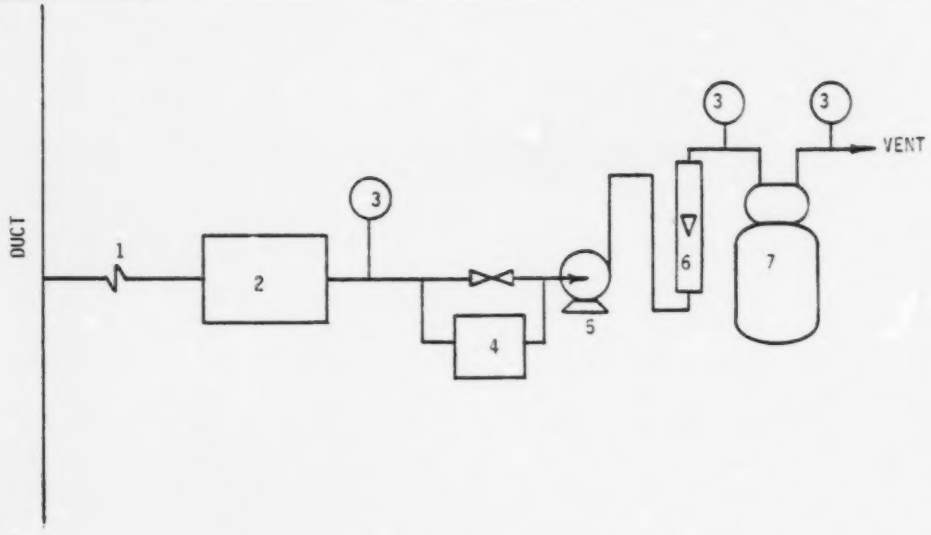

1. Sample Line
2. Sample Collection
   a. Molecular Sieve Containers
   b. Sample Pretreatment, if any
3. Dial Thermometer
4. NO_X Analyzer, Dynascience Corp. Model NX-110
5. "Air Cadet", Model 7530 leakless diaphragm pump
6. Calibrated rotameter
7. Dry Gas Meter, Rockwell Manufacturing Co., Model 175-S

Figure 1. Sampling train, configuration 1

NO_x analyzer.* This work indicated that the Dynasciences monitor used in the first two studies was biased approximately 20 percent high.† The sampling train was modified to allow use of the ThermoElectron analyzer in subsequent studies. The modified sampling train is shown schematically in Figure 2.

It should be noted that, with the Dynascience monitor, it was possible to place the monitor in the system ahead of the dry gas meter. This was not possible with the ThermoElectron monitor due to the manner in which the sample is handled by this instrument. In the ThermoElectron instrument, the bulk sample is drawn through the system by a diaphragm pump and vented to atmosphere. Within the instrument, the sample to be analyzed is drawn from the bulk sample through a capillary restriction into the analytical section of the instrument by an oil-sealed vacuum pump. Additionally, the dry gas meter could not be placed ahead of the monitor, since the large internal volume of the dry gas meter (ca. 5 liters) would act as a dilution chamber for the collection train effluent and would prevent accurate determination of the NO_x breakthrough point. Therefore, a carefully calibrated rotometer was used to determine the sampling flow rate, and the sample volumes were calculated from this flow rate and the sampling time.

The variables in the third study were the amount of molecular sieve used (13X or AW-500) and the effect of sample flow rate on NO_x collection efficiency. The molecular sieves were contained in midget stainless steel impingers. Three, four, or five impingers, each containing 19.6 g of sieves, were used. The impingers were cooled in an ice-water bath during sampling. The stack gas was dried prior to sampling by passage through three Smith-Greenberg impingers, each containing 125 mL water, followed by an empty modified Smith-Greenberg impinger. The Smith-Greenberg impingers were cooled in an ice-water bath. Sample flow rates were 2.3 or 4.3 L/min.

The variables in the fourth study included sample flow rate (0.5 or 1.0 L/min), type of molecular sieve (AW-500 or 13X), number of impingers (2 or 3, each containing 19.6 g sieve), and type of impinger (glass or stainless steel). As in the previous study, the stack gas was dried prior to sampling by passage through three Smith-Greenberg impingers,

---

* The calibration standard in the Tedlar bag served as a control sample. There was no significant difference in the analysis of the standard in the bag and directly from the cylinder.

† This bias was possibly due to an interference; however, the exact cause of the bias is not known.

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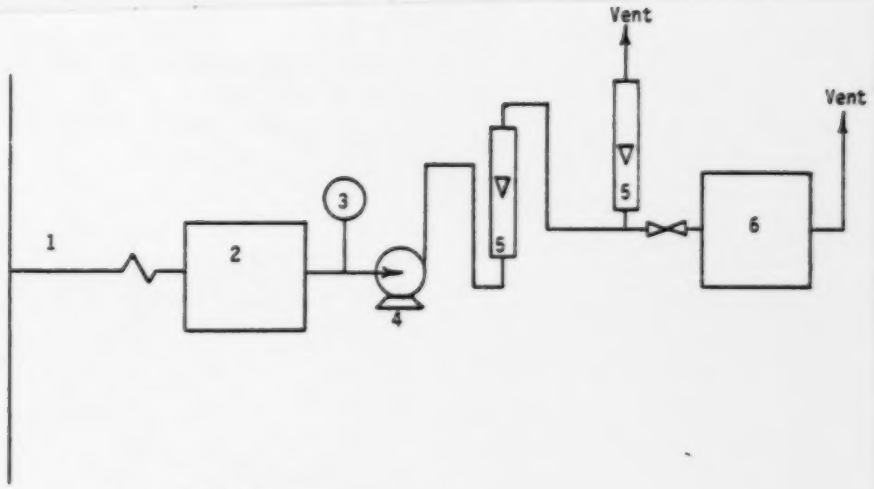

1. Sample Line
2. Sample Collection
   includes sample pretreatment
   plus molecular sieve containers
3. Dial Thermometer
4. "Air Cadet", Model 7530 leakless diaphragm pump
5. Calibrated Rotameter
6. NO_x Analyzer, ThermoElectron Model 10A

Figure 2. Sampling train, configuration 2

each containing 125 mL water, followed by an empty modified Smith-Greenberg impinger. The Smith-Greenberg impingers were cooled in an ice-water bath.

In all four field studies, the molecular sieves were quantitatively transferred to amber glass sample bottles at the conclusion of each run. In runs employing more than a single impinger, the molecular sieves from each impinger were separately bottled and appropriately labeled by run and impinger number. The molecular sieves were then sent to EPA for use in their laboratory studies on the analytical recovery portion of the method.

The reasons for selecting the above variables for study are given below:

Variable	Reason for Selection
Type of molecular sieve	To vary the pore size in NO_x collection.
Water removal prior to NO_x collection	The molecular sieves used absorb 20% of their weight of water. Therefore, to minimize reduction of the sieves' capacity to collect NO_x, the air stream was dried by condensation prior to sampling.
Geometry of molecular sieve holder	To vary residence time of NO_x in contact with the molecular sieve.
Sample flow rate	Same as above.
Amount of molecular sieve	To increase capacity for NO_x collection.
Metal vs. glass impingers	Since adsorption was believed to be involved in the collection mechanism, metal impingers were evaluated to provide more efficient cooling during collection.

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SECTION 4

RESULTS AND DISCUSSION

The initial field sampling was designed to provide preliminary information on the use of molecular sieves for NO_x collection. Work focused on the performance of particular molecular sieves, the requirements for cooling the sieve bed during sampling, any necessity for pretreating the sample prior to the sieve bed, and any effect of sieve bed configuration. The results are presented in Table 1. Duplicate runs were made utilizing Linde 13X, Linde AW-500, and Davison Grade 626 sieves (designated 13X, AW-500 and 5A in the table). Sampling rate was nominally 1 L/min. No sample pretreatment and no sieve bed cooling were employed. The stack gas contained approximately 200 ppm NO_x. In all these initial runs, using both straight glass tubes or midget impingers as sieve containers, there was immediate NO_x breakthrough in the sieve bed effluent. Single runs were then made using each type molecular sieve in glass tubes. A midget impinger train (two impingers containing 15 mL water each followed by an empty impinger) immersed in an ice-water bath was placed ahead of the molecular sieves to remove most of the water vapor from the sample. This modification produced some improvement; the best performance being with Linde AW-500 sieves and the poorest with Davison Grade 626 sieves. Placing the molecular sieves in midget impingers and immersing the sieve-containing impingers in the ice-water bath downstream of the moisture removal impingers led to further improvement, with no significant NO_x breakthrough for 8-10 minutes of sampling. A final set of runs utilizing either two or three midget impingers, each containing 19.6 g of Linde AW-500 sieves, gave breakthrough times of 16 and 24 min, respectively.

The second field study design utilized the information developed in the first study. All runs were made with a water knockout ahead of the molecular sieves. The molecular sieve-containing impingers were placed in an ice-water bath. Impingers were either glass or stainless steel, and either two or three impingers, each containing 19.6 g of molecular sieve, were used. Additionally, three sample flow rates were used. Linde AW-500 molecular sieves were used in 10 of the 12 runs; Linde 13X sieves were used in Runs 3 and 4. The sampling data are presented in Table 2. The effects of the various parameters are

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TABLE 1. SAMPLE DATA FOR USE OF MOLECULAR SIEVES FOR NO_x COLLECTION AT A POWER PLANT

Sieve	Container	H_2O KO?	Sieve cooling?	T_m (avg) °F	T Effluent from sieve container °F	Average flow rate, L/min	Stack NO_x C_o	[NO_x], ppm by Dynasciences monitor, impinger outlet, by min																
								2	4	6	8	10	12	14	16	18	20	22	24	26	28	30	32	34
13X	tube	No	No	79	--	1.25	220	40	90	90	90	90	85	85	85	90	85							
"	"	"	"	79	--	1.17	220	10	70	70	70	70	65	65	60	55	55							
AW-500	"	"	"	81	--	0.97	240	20	20	45	55	60	60	65	70	70	70							
"	"	"	"	82	--	1.09	210	10	35	50	60	65	70	75	75	80	80							
5A	"	"	"	85	84	1.06	200	15*	15	50	85	90	90	95	95	95	95							
"	"	"	"	88	90	1.05	210	5	15	70	90	90	90	95	95	95	95							
13X	impinger	"	"	88	91	1.05	210	20	70	80	80	75	75	75	75	75	80							
"	"	"	"	90	94	1.04	215	0	40	70	75	70	65	65	60	60	60							
AW-500	"	"	"	90	94	1.06	215	5*	12	24	34	37	42	50	50	48	48							
"	"	"	"	90	94	1.01	210	2	20	44	46	48	48	50	52	52	55							
5A	"	"	"	92	95	0.98	210	10	25	90	96	96	105	105	102	105	108							
"	"	"	"	93	94	1.04	210	16	45	105	115	125	125	120	125	125	125							
13X	tube	Yes	No	93	96	1.04	210	1	7	46	51	52	49	47	45	44	46							
AW-500	"	"	"	94	98	1.04	210	0	2	9	22	28	30	29	32	33	34							
5A	"	"	"	94	95	1.02	195	6	14	42	83	82	84	82	81	83	83							
13X	impinger	"	Yes	96	--	1.00	200	0	0	0	2	18	27	28	28	26	20							
"	"	"	"	96	--	1.00	200	1	0	1	4	14	21	23	23	23	23							
AW-500	"	"	"	93	95	0.99	195	0	1	0	2	12	30	35	37	38	42							
"	"	"	"	93	95	1.02	195	0	0	0	0	7	22	34	33†	34	37							
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"	"	"	"	94	96	1.05	200	0	0	0	4	13	54	72	76	75	78							
AW-500 2	impingers	"	"	81	82	1.15	200	0	4	4	2	1	2	0	2	13	22							
" 3	"	"	"	83	82	1.13	200	2	3	2	2	0	2	0	0	0	2	2	0	6	8	10	11	12
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TABLE 2. MOLECULAR SIEVES FOR NO_X COLLECTION SAMPLING DATA

Run* no.	Sieve† container	T_m average °F	T, effluent from sieve bed, °F	V_m std. $M^3 \times 10^{-3}$	Stack (NO_x) ppm	[NO_x], ppm by Dynasciences monitor, impinger outlet, by minutes																					
						1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22
1A	G-2	86	90	16.401	200	-	1	-	0	-	0	-	0	-	0	-	0	-	1	-	0	-	1	-	3	- 24/16	6 26/16
2A	SS-2	92	92	19.001	258	-	0	-	1	-	0	-	0	-	0	-	0	-	0	-	0	-	2	-	8	- 24/15	12 26/16
3A	G-2	89	91	20.249	240	-	0	-	0	-	0	-	0	-	0	-	0	-	2	-	6	-	13	-	15	- 24/13	14
4A	SS-2	87	89	17.424	260	-	0	-	0	-	0	-	0	-	0	-	0	-	1	-	1	-	3	-	8	-	14
5A	G-2	86	87	17.706	220	0	0	0	0	1	12	29	43	51	56												
6A	M-2	85	88	17.359	220	0	0	0	0	0	12	30	38	48	51						8						
7A	G-3	85	85	25.830	210	0	0	0	0	0	0	0	0	0	4	12	21	28	33	35							
8A	M-3	84	81	26.119	235	0	0	0	0	0	0	0	0	2	8	17	25	30	34	26							
9A	G-3	82	85	25.650	210	0	0	0	1	2	9	18	29	38	44												
10A‡	M-3	-	-		-																						
11A	M-2	83	84	14.173	210	0	0	0	0	0	2	9	15														
12A	G-2	83	84	13.985	240	0	0	0	0	0	0	10	24														

Notes:

* All runs made with 19.6 grams of molecular sieve per impinger.
All runs made with AW-500 sieves except 3 and 4 utilized 13X sieves.
All runs made with a water knockout and ice water cooling on the impinger.

† Midget impingers: G dep[illegible]ass, SS denotes stainless steel, the arabic numeral denotes the number o[illegible] series.

‡ Run aborted-high flow rate through three metal impingers could not be obtained.

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examined in Table 3. Volume of NO_x absorbed at NO_x breakthrough was generally directly proportional to sample flow rate, as would be expected. This relationship is shown by comparing the rank of the AW-500 runs using two impingers with the order of increasing flow rate. As would also be expected, the time to NO_x breakthrough generally decreased as the sample flow rate increased. Finally, collection efficiency was generally better with stainless steel impingers than with glass impingers, other variables being equal. This is indicated by the relative ranking of the runs given in Table 3. As can be seen in each set of runs (1-2, 3-4, etc.) allowing direct comparison of impinger construction materials, in every case but one, the stainless steel impinger is of lower rank than the glass impinger, i.e., the volume of gas sampled at NO_x breakthrough was larger for the stainless steel impinger. This was interpreted to reflect enhanced cooling of the sieve bed with the metal impingers.

The third field study further evaluated the effect of sample flow rate and the amount of molecular sieve on NO_x collection efficiency. The sample pretreatment system was modified. In place of midget impingers, three Smith-Greenberg impingers containing 125 mL water each, followed by an empty modified Smith-Greenberg impinger, were used for a water vapor knockout. The NO_x collection efficiency was assessed in two ways: time to NO_x breakthrough and volume of NO_x absorbed at breakthrough. The run data for this study are shown in Table 4; the molecular sieve effluent gas NO_x concentration with time in Table 5; and the volume of NO_x absorbed per gram of sieve at breakthrough in Table 6.

The time to NO_x breakthrough data in Table 5 show that sampling efficiency increased with decreasing flow rate and time to NO_x breakthrough increased with increasing amount of molecular sieve. These same trends are shown by the data in Table 6, displaying volume of NO_x absorbed per gram of molecular sieve. Thus, with both types of molecular sieve, the sampling efficiency increased with increasing amount of molecular sieve and with decreasing sample flow rate.

There was some concern that the use of the Smith-Greenberg impingers with their larger water volumes would lead to low NO_x values in the sample gas due to dissolution of NO and/or NO_2 in the water. A brief laboratory experiment was conducted to determine if a significant absorption of NO occurred in the Smith-Greenberg impinger system used as the water knockout. Cylinder gas containing NO in nitrogen was passed through the knockout system at 1 L/min, and the effluent gas NO concentration was determined using a ThermoElectron Model 10A chemiluminescent analyzer. Periodically, the gas stream was diverted directly to the analyzer to check for instrument drift. The data from this experiment are presented

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TABLE 3. EFFECT OF SAMPLE FLOW RATE AND THE AMOUNT OF MOLECULAR SIEVE ON NO_x COLLECTION EFFICIENCY

Run no.	Mole sieve type	No. of impingers	Type of impinger	Average flow rate, L/min.	$[NO_x]$, ppm stack	Time to NO_x breakthrough, min.	Volume to breakthrough, mL NO_x	Order of increasing flow rate: All runs	Order of increasing flow rate: Runs with 2 impingers AW-500	Rank NO_x breakthrough volume: All runs	Rank NO_x breakthrough volume: Runs with 2 impingers AW-500
1A	AW-500	2	G	0.631	200	20.0	2.52	1	1	8	3
2A	"	"	SS	0.731	258	18.0	3.39	3	2	4	1
3A	13 x	"	G	0.844	240	15.0	3.04	4	N.A.	5	
4A	"	"	SS	0.726	260	18.0	3.40	2	N.A.	3	
5A	AW-500	"	G	1.771	220	5.5	2.14	9	5	11	6
6A	"	"	SS	1.790	220	5.5	2.17	10	6	10	5
7A	"	3	G	1.722	210	10.0	3.62	7	N.A.	2	
8A	"	"	SS	1.741	235	9.5	3.89	8	N.A.	1	
9A	"	"	G	2.565	210	5.5	2.96	11	N.A.	6	
10A	"	"	SS	*	190	—	—	—	N.A.	—	
11A	"	2	SS	0.886	210	13.0	2.42	6	4	9	4
12A	"	"	G	0.874	240	13.0	2.73	5	3	7	2

* Run absorbed - desired flow rate could not be obtained.

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TABLE 4. MOLECULAR SIEVE SAMPLING FOR NO_X RUN DATA

Run no.	No.[a] of impingers	Type of[b] sieve	Sampling rate, L/min	Stack[c] NO_X ppm	T, effluent From Sieve Bed	Volume NO_X absorbed[d] at breakthrough, mL	Time to breakthrough, min.
3B	3	13X	4.3	320	89	9.6	6
4B	3	"	2.3	335	85	11.6	14
5B	4	"	4.3	340	86	10.2	6
6B	4	"	2.3	345	83	24.6	30
7B	5	"	4.3	335	82	18.7	12
8B	3	AW-500	4.3	335	84	10.1	6
9B	3	"	2.3	335	81	16.2	20
10B	3	"	4.3	320	86	9.6	6
11B	4	"	4.3	340	87	16.1	10
12B	5	13X	2.3	350	83	33.0	40
13B	4	AW-500	2.3	320	81	22.8	30
14B	5	"	4.3	335	86	27.4	18
15B	5	"	2.3	335	82	30.0	38

a Stainless steel miniature impingers. Impingers cooled in an ice-water bath on all runs.

b. Each impinger contained 19.6 g of the indicated sieve.

c Stack and impinger train effluent NO_X concentration determined with a ThermoElectron Model 10A Chemiluminescent analyzer. Stack gas was conditioned prior to sampling and analysis by passage through three water-containing Smith-Greenberg impingers followed by an empty impinger, all in an ice bath.

d NO_X breakthrough is the point where the impinger train effluent NO_X concentration exceeds 2.0 ppm and increases markedly with time.

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TABLE 5. IMPINGER TRAIN EFFLUENT NO_x CONCENTRATION WITH TIME

Run no.	Stack NO_x conc. ppm	Time, min																									
		2	4	6	8	10	12	14	16	18	20	22	24	26	28	30	32	34	36	38	40	42	44	46	48	50	52
3B	320	0.4	1.3	1.9	3.3	7.8	26.0	35.0	36.0	34.0																	
4B	335	0.5	0.4	0.5	0.5	0.9	1.2	1.5	2.2	4.0	9.2	15.0	16.5	16.5	16.5	16.5											
5B	340	0.4	0.6	1.4	2.4	5.0	15.5	28.0	28.0	28.0	28.0																
6B	345	0.4	0.4	0.4	0.4	0.4	0.4	0.4	0.4	0.4	0.4	0.4	0.5	0.9	1.2	1.8	3.2	6.8	10.0	9.5	9.0	9.0	9.0	9.0			
7B	335	0.6	0.6	0.7	1.1	1.4	2.0	3.8	12.5	24.0	23.0	22.0	22.0														
8B	335	0.9	0.9	1.3	11.0	34.0	40.0	43.0	45.0	47.0																	
9B	335	1.2	1.2	1.2	1.2	1.2	1.2	1.2	1.2	1.2	1.5	4.8	13.0	18.0	20.0	20.0											
10B	320	1.4	1.4	1.5	3.3	21.5	44.0	46.0	50.0																		
11B	340	1.8	1.9	1.9	1.9	1.9	3.2	19.0	34.0	41.0	43.0	46.0	45.0														
12B	350	0.2	0.2	0.1	0.1	0.1	0.1	0.2	0.2	0.3	0.3	0.4	0.3	0.3	0.3	0.3	0.3	0.1	0.1	0.2	1.0	3.0	6.2	7.0	7.0	6.9	6.6
13B	320	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	6.0	9.0	11.5	14.0	14.0	14.5	14.5	15.0			
14B	335	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.2	2.0	13.0	21.5	24.0	24.0	24.0												
15B	335	1.0	1.0	1.0	1.0	1.0	1.2	1.2	1.2	1.2	1.2	1.3	1.4	1.4	1.6	1.6	1.7	1.7	1.8	2.0	4.5	9.0	11.0	11.5	12.0	12.0	

Vertical bar denotes breakthrough point, arbitrarily defined as a 2ppm effluent NO_x concentration.

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TABLE 6. VOLUME OF NO_x ABSORBED AT BREAKTHROUGH PER GRAM OF SIEVE

Run no.	Sieve type	Sample flow rate L/min	No. of impingers	NO_x absorbed mL/g sieve
3B	13X	4.3	3	0.163
4B	"	2.3	3	0.197
5B	"	4.3	4	0.130
6B	"	2.3	4	0.314
7B	"	4.3	5	0.191
12B	"	2.3	5	0.337
8B	AW-500	4.3	3	0.172
10B	"	4.3	3	0.163
9B	"	2.3	3	0.276
11B	"	4.3	4	0.205
13B	"	2.3	4	0.291
14B	"	4.3	5	0.280
15B	"	2.3	5	0.306

in Table 7. At both NO concentrations, the data show that the water in the knockout becomes saturated in 10-15 min, and the knockout effluent had the same NO concentration as the inlet gas. This point should be examined on field samples containing NO + NO_2. If equilibrium is obtained, a requirement would have to be included in the field sampling method to purge the knockout for a specified time period, possibly 20 minutes, prior to the initiation of sampling for NO_x. If equilibrium is not obtained (due to absorption of NO_2), the contents of the water knockout system would have to be analyzed along with the molecular sieves.

A final field study was conducted in which run parameters (amount of sieve, sample flow rates, and type of impinger) were close to values planned for a tentative method. Specifically, either two or three impingers were used in series at sample flow rates of 0.5 or 1.0 L/min. Both stainless steel and glass impingers were used. Sample conditions were as described in the third study, except that the knockout impingers were purged with stack gas for 30 minutes prior to sampling. The molecular sieve impingers were cooled in an ice-water bath. The run data are shown in Table 8, and the impinger train effluent NO_x concentration with time for each run is shown in Table 9. The only cases where NO_x breakthrough occurred were on runs utilizing two impingers in conjunction with a sample flow rate of 1.0 L/min, and the worst case was breakthrough in 26 min. It should also be mentioned that for purposes of comparison in this study, NO_x breakthrough was arbitrarily defined as NO_x concentration of 2 ppm in the impinger train effluent. At the stack NO_x concentrations encountered in this study, the 2 ppm breakthrough point represents less than 1 percent loss of sampling efficiency.

The field data obtained indicate that molecular sieves can be used to efficiently collect NO_x emissions from utility boilers, and a method based upon such use would have the advantage of providing an integrated stack NO_x concentration over a 20- to 40-min sample period, compared to the current practice of obtaining three 15-second duration evacuated flask samples over a 20-min period, as specified in EPA Method 7.

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TABLE 7. NO ABSORPTION WHEN USING A WATER KNOCKOUT TRAP

Run no.	Time, min	NO, ppm * Without H_2O knockout	With H_2O knockout
1	0	262	
	5		190
	10		258
	15		260
	20	262	
	25		262
	30		262
	35		262
	40	262	
	45		262
2	0	265	
	1		80
	2		90
	3		105
	4		130
	5		184
	6		220
	7		242
	8		256
	9		260
	10		260
	11	263	
	12		262
	13		262
	14		265
	15		265
3	0	2400	
	5		1600
	10		2300
	15		2300
	20	2300	
	25		2300
	30		2300

* Feed: Runs 1 and 2 - 265 ppm NO in N_2, 1.0 L/m.
Run 3 - 2400 ppm NO in N_2, 1.0 L/m.

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TABLE 8. MOLECULAR SIEVE SAMPLING FOR NO_X RUN DATA

Run no.	Type[a] of impingers	No. of impingers	Type[b] of sieve	Sampling rate, L/min	Sample T before sieves, °F	Sample T after sieves, °F	Stack[c] NO_X conc., ppm	Volume NO_X[d] absorbed, mL	Volume NO_X absorbed[e] at breakthrough, mL
2C	SS	2	AW-500	0.5	54	52	222	4.4	---
3C	SS	2	AW-500	0.5	56	55	202	4.0	---
4C	SS	3	AW-500	0.5	52	49	220	4.4	---
5C	SS	3	AW-500	0.5	53	50	205	4.1	---
6C	SS	2	13X	0.5	55	52	203	4.1	---
7C	SS	2	13X	0.5	57	54	210	4.2	---
8C	SS	3	13X	0.5	59	56	213	4.3	---
9C	SS	3	13X	0.5	60	57	208	4.2	---
10C	SS	2	13X	1.0	62	58	216	8.2	6.9
11C	SS	2	13X	1.0	61	59	230	8.3	7.8
12C	SS	3	13X	1.0	56	51	255	10.2	---
13C	SS	3	13X	1.0	53	49	241	9.6	---
14C	SS	2	AW-500	1.0	57	54	245	6.9	6.4
15C	SS	2	AW-500	1.0	56	54	248	7.4	6.9
16C	SS	3	AW-500	1.0	56	53	240	9.6	---
17C	SS	3	AW-500	1.0	57	53	250	10.0	---
18C	G	2	AW-500	1.0	65	65	250	8.0	7.5
19C	G	2	AW-500	1.0	64	64	250	8.0	7.5
20C	G	2	13X	1.0	61	61	250	8.8	8.5
21C	G	2	13X	1.0	64	63	260	8.8	8.3
22C	G	3	13X	1.0	64	63	240	9.6	---
23C	G	3	13X	1.0	65	64	250	10.0	---
24C	G	3	AW-500	1.0	64	63	260	10.4	---
25C	G	3	AW-500	1.0	65	64	275	11.0	---
26C	G	2	AW-500	0.5	66	65	255	10.2	---
27C	G	2	AW-500	0.5	64	63	240	9.6	---
28C	G	2	13X	0.5	68	67	250	10.0	---
29C	G	2	13X	0.5	69	68	260	10.4	---
30C	G	3	AW-500	0.5	80	79	280	11.2	---
31C	G	3	AW-500	0.5	82	81	250	10.0	---
32C	G	3	13X	0.5	81	80	260	10.4	---
33C	G	3	13X	0.5	80	80	250	10.0	---

[a] Two types of miniature ("Midget") impingers: SS = stainless steel and G = glass.

[b] Each impinger contained 19.6 g of the indicated sieve.

[c] Stack and impinger train effluent NO_X concentration were determined with a ThermoElectron Model 10A Chemiluminescent Analyzer. Stack gas was conditioned prior to sampling and analysis by passage through three water-containing Smith-Greenberg impingers followed by an empty impinger, all in an ice bath.

[d] Volume of NO_X is at temperature and pressure given in table.

[e] NO_X breakthrough is the point where the impinger train effluent NO_X concentration exceeds 2.0 ppm and increases markedly with time. Where no value is given in this column, breakthrough was not obtained after 40 minutes of run time. The volume given is at temperature and pressure given in table.

TABLE 9. IMPINGER TRAIN EFFLUENT NO_x CONCENTRATION WITH TIME

Run no.	Stack NO_x conc., ppm	Time, min																			
		2	4	6	8	10	12	14	16	18	20	22	24	26	28	30	32	34	36	38	40
2C	222	0.4	0.3	0.3	0.3	0.3	0.3	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2
3C	202	0.7	0.5	0.4	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3
4C	220	0.4	0.3	0.2	0.2	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
5C	205	—	0.3	0.2	0.2	0.2	0.2	0.2	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
6C	203	0.7	0.3	0.2	0.2	0.2	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
7C	210	0.5	0.3	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2
8C	213	0.5	0.4	0.4	0.4	0.3	0.3	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2
9C	208	—	0.4	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3
10C	216	0.4	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.4	0.6	0.8	2.0 |	*3.5	5.5	7.0	
11C	230	0.4	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.5	0.8	1.5	* 3.2		
12C	255	0.2	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
13C	241	0.2	0.2	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
14C	245	0.2	0.2	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.4 |	2.0*						
15C	248	0.4	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	1.4	* 5.4					
16C	240	—	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
17C	250	0.2	0.2	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.3
18C	250	0.3	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.4	* 2.1				
19C	250	0.4	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.4	1.2	* 4.7				
20C	250	0.5	0.4	0.4	0.4	0.4	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.4	0.8	1.8	* 2.7	(35 min)	
21C	260	0.4	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.4	0.6	1.1	*2.3			
22C	240	0.2	0.2	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
23C	250	0.2	0.2	0.2	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
24C	260	0.3	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.3
25C	275	0.3	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.4
26C	255	0.5	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3
27C	240	0.5	0.4	0.3	0.4	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3
28C	250	0.5	0.4	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3
29C	260	0.7	0.5	0.4	0.4	0.4	0.4	0.4	0.4	0.4	0.4	0.4	0.4	0.4	0.4	0.4	0.4	0.4	0.4	0.4	0.4
30C†	280	0.8	0.7	0.7	0.6	0.6	0.6	0.7	0.7	0.7	0.7	0.7	0.7	0.7	0.7	0.8	0.8	0.8	0.8	0.8	0.9
31C†	250	1.4	1.2	1.2	1.2	1.2	1.2	1.2	1.2	1.2	1.2	1.3	1.3	1.3	1.3	1.4	1.4	1.4	1.5	1.5	1.5
32C†	260	1.9	1.8	1.7	1.7	1.7	1.7	1.7	1.7	1.8	1.8	1.8	1.8	1.8	1.8	1.8	1.8	1.9	1.9	1.9	1.9
33C†	250	2.4	2.2	2.1	2.1	2.1	2.1	2.1	2.1	2.1	2.1	2.1	2.1	2.1	2.1	2.1	2.1	2.1	2.1	2.1	2.1

* Vertical bar denotes breakthrough point, arbitarily defined as exceeding 2 ppm effluent, NO_x concentration (does not apply to Run 33C).

† These samples were obtained with a different sampling pump (other pump failed). Higher readings were presumed to be caused by zero drift in the monitor.

BEGIN LAST LINE OF TEXT
3/8"
EPA-287 (Gin.) (4-76)
PAGE NUMBER
20
BOTTOM OF IMAGE AREA
OUTSIDE DIMENSION FOR TABLES AND ILLUSTRATIONS
BEGIN FIRST LINE OF TEXT HERE
DROPPED HEAD; BEGIN SECTIONS HERE
TYPING GUIDE SHEET
CENTER OF PAGE
TOP OF IMAGE AREA